UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
NATIONAL EXPOSURE RESEARCH LABORATORY
RESEARCH TRIANGLE PARK, NC 27711

OFFICE OF
RESEARCH AND DEVELOPMENT

April 4, 2018

Mr. Clark Friese, Assistant Commissioner
New Hampshire Department of Environmental Services (NH DES)
29 Hazen Drive
P.O. Box 95
Concord, New Hampshire 03301

Dear Mr. Friese:

I am pleased to provide you with this initial laboratory report of perfluorocarboxylate (PFCA) concentrations in solid samples (char and soil). This report is in response to your request of June 22, 2017 asking for laboratory assistance analyzing per- and polyfluoroalkyl substances (PFAS) in environmental samples. PFCAs are a subset of PFAS. This report relates to solid matrix samples sent to our Athens Lab that included three tower char and another three soil samples. We understand that these samples were collected by New Hampshire Department of Environmental Services (NH DES), on August 23, 2017 and September 8, 2017, respectively. These samples were received at our Athens Lab by Dr. John Washington who was also responsible for their lab processing.

It is our understanding that this information was requested by NH DES to help in their ongoing investigation into the presence of PFAS in the environment near manufacturing facilities of interest. This request relates to our research capabilities and interests applying targeted and non-targeted analysis methods for discovery of the nature and extent of PFAS environmental occurrence associated with industrial releases. The current report is limited to targeted results only. Our non-targeted work requires considerable post-processing manual effort and therefore will lag the targeted results.

EPA continues to develop analytical methods for many PFAS compounds in various media including some of those included in this report. The data enclosed provides information related to the concentration of certain PFAS in the media sampled. In this report we do not interpret exposure or risk from these values. EPA does not currently have health based standards, toxicity factors or associated risk levels for PFAS, other than perfluoroctanoic acid (PFOA), perfluoroocatane sulfonate (PFOS), and perfluorobutanesulfonic acid (PFBS). Therefore, while the data presented indicate the presence of PFCA, no conclusions can be made related to human or environmental exposure and risk.

Thank you for inviting us to be part of this effort that helps to further both EPA's and New Hampshire's understanding of an important issue in the state. This is just one of many Agency efforts that demonstrate EPA's commitment to cooperative federalism.

April 4, 2017

The results presented in the attachment represent the work of many within ORD's National Exposure Research Laboratory. Our technical experts include Drs. John Washington, Mark Strynar, Andy Lindstrom, Seth Newton, Thomas Jenkins, and James McCord. Our Quality Assurance team includes Sania Tong-Argao and Brittany Stuart. Management support and coordination has been provided by Drs. Timothy Buckley, Myriam Medina-Vera, Jack Jones, Adam Biales, and Brian Schumacher.

If you have any questions or concerns, do not hesitate to contact me at (919) 541-2106 or via email at watkins.tim@epa.gov or Tim Buckley at (919) 541-2454 or via email at buckley.timothy@epa.gov. I look forward to our continued work together.

Sincerely,

Timothy H. Watkins
Director

Attachment

cc: Meghan Cassidy, USEPA, Region 1
Deb Szaro, USEPA, Region 1
Jeff Morris, USEPA OPPT
Betsy Behl, USEPA, OW
Peter Grevatt, USEPA, OW
Andy Gillespie, USEPA, ORD
Timothy Buckley, USEPA, ORD

Summary of Methods and Results

New Hampshire Department of Environmental Services (NH DES), in coordination with Region 1, requested ORD's technical support in analyzing PFAS in environmental samples potentially impacted by industrial sites within the state. NH DES assumed responsibility for the collection of samples and their shipment to our laboratories. ORD was responsible for sample extraction and analysis of PFAS. We are providing the results of our analysis as they become available. This is our first report.

The current report includes results for char (n=3) and soil (n=3) samples that were sent to and analyzed under the direction of Dr. John Washington within our Athens Lab. Samples were collected in containers provided by NH DES and shipped to EPA. Thirteen PFCA analytes (Table 1) were analyzed using methods described within an approved Quality Assurance Project Plan (QAPP)[1] and that have been generally described in Rankin et al., 2015.[2] In brief, each sample was divided into three ~1 g aliquots and extracted and analyzed in triplicate. Extracts were analyzed by liquid chromatography / mass spectrometry (Waters Acquity UPLC coupled to a Waters Quattro Premier XE tandem) and quantified using mass-labeled internal standards. These analyses were performed on samples, process blanks, and check standards using internal-calibration curves for quantitation. The mean value of the triplicate analysis is reported. Reported results are based on the identification and quantification of analytes using certified standards (i.e., targeted analysis).

Some of the sample extract required dilution so that concentrations were within the acceptable range of the calibration curve. The reported results have been adjusted for each dilution factor and flagged accordingly in Table 2.

Table 1. Summary of Reported Perfluorocarboxylates

PFCA	Compound Name	Acronym	CAS Number
C4	Perfluorobutanoic acid	PFBA	375-22-4
C5	Perfluoropentanoic acid	PFPeA	2706-90-3
C6	Perfluorohexanoic acid	PFHxA	307-24-4
C7	Perfluoroheptanoic acid	PFHpA	375-85-9
C8	Perfluorooctanoic acid	PFOA	335-67-1
C9	Perfluorononanoic acid	PFNA	375-95-1
C10	Perfluorodecanoic acid	PFDA	335-76-2
C11	Perfluoroundecanoic acid	PFUnDA	2058-94-8
C12	Perfluorododecanoic acid	PFDoDA	307-55-1
C13	Perfluorotridecanoic acid	PFTrDA	72629-94-8

[1] Strynar, M.; Washington, J.; Lindstrom, A.; Henderson, W. 2017. Quality Assurance Project Plan: Non-Targeted Analyses of Per- and Polyfluoroalkyl Substances (PFAS) for New Hampshire Department of Environmental Services (NHDES). D-EMMD-PHCB-015-QAPP-01.

[2] K. Rankin, S. A. Mabury, T. M. Jenkins, J. W. Washington, A North American and global survey of perfluoroalkyl substances in surface soils: Distribution patterns and mode of occurrence. Chemosphere 161, 333-341 (2015).

C14	Perfluorotetradecanoic acid	PFTeDA	376-06-7
C16	Perfluorohexadecanoic acid	PFHxDA	67905-19-5
C18	Perfluorooctadecanoic acid	PFOcDA	16517-11-6

Targeted results are provided in Table 2 below for 13 perfluorocarboxylates that range from C4 through C18. Results are reported in mass of PFAS per unit mass of dry solid. Please note that the units are reported in µg/g for char samples and pg/g for soil samples. Precision of our measurements was estimated by the relative standard deviation (RSD) of triplicate extractions/analyses. For char across all of the compounds, the median RSD was 12.9% and ranged from 1.6% to 48.8%. Similarly for soils, the median RSD was 19.2% and ranged from 1.2 to 51.6%. The values reported for target analytes all exceeded levels detected in process blanks ($p \leq 0.05$) and were corrected for any low detections in process blanks. No field blanks were provided or analyzed; however, quality control check standards were analyzed at varying concentrations throughout the analysis to ensure that measurements at varying points of the calibration range were within quality control specifications. Recovery of these standards ranged from 79.3% to 118% which was within our ± 30% criteria for acceptability.

As was expected, the measured perfluorocarboxylates tended to occur at higher concentrations in the char samples than the soil with char results ranging from <LOD (limit of detection) to 1430 µg/g. Soil results were in the pg/g range and varied from <LOD to 7420 pg/g. Among the char, PFCA concentrations were consistently higher at "QX Tower" followed by the "MS Tower," and lowest for the "MA Tower." Similarly for soil, PFCA concentrations generally trended "EPAORDS1" > "EPAORDS2" > "EPAORDS3." Across both matrices, the highest concentration was consistently observed for C8 (PFOA).

Table 2. Concentration of Perfluorocarboxylates Measured in New Hampshire Samples

PFCA	Sample ID	Matrix	Conc.	Unit	Flag(s)*
C4	MS Tower	Char	3.13	µg/g	D2
C5	MS Tower	Char	4.41	µg/g	D2
C6	MS Tower	Char	15.1	µg/g	D2
C7	MS Tower	Char	7.60	µg/g	D2
C8	MS Tower	Char	439	µg/g	D3
C9	MS Tower	Char	4.25	µg/g	D2
C10	MS Tower	Char	9.44	µg/g	D2
C11	MS Tower	Char	4.55	µg/g	D2
C12	MS Tower	Char	9.12	µg/g	D2
C13	MS Tower	Char	5.07	µg/g	D2
C14	MS Tower	Char	10.8	µg/g	D2
C16	MS Tower	Char	7.95	µg/g	D2
C18	MS Tower	Char	--	µg/g	D2, <LOD
C4	QX Tower	Char	20.1	µg/g	D2
C5	QX Tower	Char	41.7	µg/g	D2
C6	QX Tower	Char	122	µg/g	D2
C7	QX Tower	Char	71.5	µg/g	D2

PFCA	Sample ID	Matrix	Conc.	Unit	Flag(s)*
C8	QX Tower	Char	1430	µg/g	D3
C9	QX Tower	Char	36.2	µg/g	D2
C10	QX Tower	Char	74.8	µg/g	D2
C11	QX Tower	Char	52.2	µg/g	D2
C12	QX Tower	Char	87.5	µg/g	D2
C13	QX Tower	Char	62.9	µg/g	D2
C14	QX Tower	Char	79.3	µg/g	D2
C16	QX Tower	Char	28.4	µg/g	D2, <LOQ
C18	QX Tower	Char	3.14	µg/g	D2, <LOQ
C4	MA Tower	Char	--	µg/g	D2, <LOD
C5	MA Tower	Char	--	µg/g	D2, <LOD
C6	MA Tower	Char	0.212	µg/g	D2, <LOQ
C7	MA Tower	Char	--	µg/g	D2, <LOD
C8	MA Tower	Char	2.55	µg/g	D2, <LOQ
C9	MA Tower	Char	--	µg/g	D2, <LOD
C10	MA Tower	Char	--	µg/g	D2, <LOD
C11	MA Tower	Char	--	µg/g	D2, <LOD
C12	MA Tower	Char	0.100	µg/g	D2, <LOQ
C13	MA Tower	Char	0.095	µg/g	D2, <LOQ
C14	MA Tower	Char	0.193	µg/g	D2
C16	MA Tower	Char	0.423	µg/g	D2
C18	MA Tower	Char	0.357	µg/g	D2, <LOQ
C4	EPAORDS1	Soil	194	pg/g	UD
C5	EPAORDS1	Soil	389	pg/g	UD, <LOQ
C6	EPAORDS1	Soil	1270	pg/g	UD
C7	EPAORDS1	Soil	615	pg/g	UD
C8	EPAORDS1	Soil	7420	pg/g	D1, <LOQ
C9	EPAORDS1	Soil	240	pg/g	UD
C10	EPAORDS1	Soil	238	pg/g	UD, <LOQ
C11	EPAORDS1	Soil	90.0	pg/g	UD, <LOQ
C12	EPAORDS1	Soil	--	pg/g	UD, <LOD
C13	EPAORDS1	Soil	--	pg/g	UD, <LOD
C14	EPAORDS1	Soil	--	pg/g	UD, <LOD
C16	EPAORDS1	Soil	--	pg/g	UD, <LOD
C18	EPAORDS1	Soil	--	pg/g	UD, <LOD
C4	EPAORDS2	Soil	--	pg/g	UD, <LOD
C5	EPAORDS2	Soil	--	pg/g	UD, <LOD
C6	EPAORDS2	Soil	175	pg/g	UD, <LOQ
C7	EPAORDS2	Soil	540	pg/g	UD
C8	EPAORDS2	Soil	6950	pg/g	UD
C9	EPAORDS2	Soil	--	pg/g	UD, <LOD
C10	EPAORDS2	Soil	34.3	pg/g	UD, <LOQ

PFCA	Sample ID	Matrix	Conc.	Unit	Flag(s)*
C11	EPAORDS2	Soil	--	pg/g	UD, <LOD
C12	EPAORDS2	Soil	--	pg/g	UD, <LOD
C13	EPAORDS2	Soil	--	pg/g	UD, <LOD
C14	EPAORDS2	Soil	--	pg/g	UD, <LOD
C16	EPAORDS2	Soil	--	pg/g	UD, <LOD
C18	EPAORDS2	Soil	--	pg/g	UD, <LOD
C4	EPAORDS3	Soil	--	pg/g	UD, <LOD
C5	EPAORDS3	Soil	18.9	pg/g	UD, <LOQ
C6	EPAORDS3	Soil	56.1	pg/g	UD, <LOQ
C7	EPAORDS3	Soil	165	pg/g	UD, <LOQ
C8	EPAORDS3	Soil	1140	pg/g	UD
C9	EPAORDS3	Soil	21.4	pg/g	UD, <LOQ
C10	EPAORDS3	Soil	19.4	pg/g	UD, <LOQ
C11	EPAORDS3	Soil	5.70	pg/g	UD, <LOQ
C12	EPAORDS3	Soil	--	pg/g	UD, <LOD
C13	EPAORDS3	Soil	--	pg/g	UD, <LOD
C14	EPAORDS3	Soil	--	pg/g	UD, <LOD
C16	EPAORDS3	Soil	--	pg/g	UD, <LOD
C18	EPAORDS3	Soil	--	pg/g	UD, <LOD

* Flags defined:
UD = undiluted
D1 = 10-fold dilution
D2 = 10^4-fold dilution
D3 = 10^5-fold dilution
<LOQ = Less than limit of quantitation (defined as exceeding process blanks $p>0.05$ but ≤ 0.001)
<LOD = Less than limit of detection (defined by $p \leq 0.05$)